

## Decoding variant for dilatometric measurements of metal samples heating and cooling

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**Abstract.** Dilatometric measurements in the form of sample sizes registration with temperature changes are widely used in the practice of physical materials science. In recent decades, the hardware component of these measurements has substantially changed with a practically unchanged version of their use as applied to the characteristics of various phase transformations. The basic point, in this case, is the identification of the critical points temperature for the ‘start/finish’ transformations based on the analysis of dilatograms deviations from a tangents carried out in the temperature intervals near the break observed on the obtained experimental curves. However, in most cases, this information has a framework character due to the presence of deficiencies in the critical points temperatures determining. In some cases, to compensate for these shortcomings, results of the changes analysis in the first derivative of dilatometric curves are additionally used. A series of works carried out by the authors is studying the features at changing the temperature coefficient of linear expansion true values. An algorithm for obtaining this spectrum is prescribed.

### 1. Introduction

Dilatometric measurements are used to search for temperature ranges of structural and phase transformations in materials [1, 2]. For this, a technique is used to register the beginning and end of transformations. The determination of temperature points is performed by crossing the tangents drawn in the area of dilatometric curves inflections. The curves are obtained during the experiment by recording the thermal expansion/contraction of samples linear dimensions [3].

The technique is simple and clear. However, the technique neglects some experimental curves features. For example, the nonlinearity of dilatograms at inflection points, as well as the change in their slope within and outside the temperature ranges of the analyzed structural changes. In this case, part of the information about transformations features has been lost.

An additional method for decoding dilatograms is the analysis of the temperature coefficient of linear expansion of samples (LTEC) curves, obtained by differentiating the experimental curve. The most widely used is the calculation of the LTEC values relative to room temperature (20 °C) [4]. It is integral information on the variation of the thermal expansion coefficient within the temperature ranges from 20 °C to the upper temperatures. Unfortunately, these calculations used in the design calculations for building and engineering parts are accompanied by significant distortions of the ‘true’



values of thermal expansion coefficient [4]. It cannot unambiguously reflect the occurrence of structure transformations in materials upon thermal exposure to them.

The practice of using the 'true' values of the thermal expansion coefficient (true) [3, 4], obtained by numerical differentiation based on two adjacent points of experimental dilatograms, showed a higher information content in the unambiguous reflection of the desired structural and phase transformations occurring in the material of the samples subjected to heat. This attracted attention to the determination of the true values of LTEC (true) [6–11].

However, there is a need for a background 'zero' line, against which dilatometric effects can be distinguished. Similar lines are widely used in chromatography when working with curves that have various kinds of peaks that reflect the course of various processes. In the case of dilatometry, a variant of the background 'zero' line of LTEC heating ('DL-BACKGROUND') was proposed in a previous work [7]. These values are obtained by numerically differentiating the analyzed experimental dilatograms.

Namely, the partial contribution of only the thermal vibrations of atoms is used. This is a monotonically changing process of the same sign within the temperature range of exposure to the sample. In this case, the effects of structural and phase transformations accompanying the true thermal change in the size of the sample are discarded.

In turn, it is taken into account that the monotonicity of the LTEC curve is preserved only in the temperature limits of the existence of one type crystal lattice matrix structure in the investigated material.

The monotonic component of the true LTEC curves was allowed to be interpreted by individual logarithmic curves. It is this dependence, based on purely external signs, that most successfully reflects the change in the course of the temperature curves of the true values of the thermal expansion coefficient for pure substances of the periodic system. This fact is formulated on the basis of the experimental data contained in the work of Novikova [4]. Moreover, the concept of vacancy and quantum-mechanical corrections taking place above 500 °C and below room temperature, respectively, was introduced [5].

## 2. Research Methodology

The methodology allows one to interpret the monotonicity of the LTEC temperature variation in graphical form by a line constructed on one experimental point. And in the digital version, the technique allows you to display the equations of the logarithmic dependence with the pre-logarithmic factor ( $A_{ln}$ ). The value of the factor ( $A_{ln}$ ) is functionally related to the melting temperature of the analyte [4, Figure 2a]. This suggests the existence of a functional relationship between the energy of interaction of atoms in the structure of the analyte with the value of the selected number ( $A_{ln}$ ).

The presence of polymorphism suggests the use of two similar curves as applied to modifications of crystal lattices. Moreover, the available experimental data suggest that for the iron-carbon system the ratio of the coefficients ( $A_{ln}$ ) for the ferritic and austenitic components is in the ratio of about two to three, respectively.

The combination of two monotonous phase segments with an inserted closing straight line allows us to obtain a continuous 'total' broken curve of the true values of LTEC – 'LTEC' – BACKGROUND (total). Using the numerical integration of the obtained total curve 'LTEC – BACKGROUND (total)' allows us to obtain a curve with the dimension of the experimental dilatogram reflecting the 'expected' thermal change in sample size in its pure form. It was decided to use this curve directly as the background 'zero' line of the experimental dilatogram – 'DL-BACKGROUND'. Accordingly, any deviations from this line can be considered as dilatometric effects [12].

## 3. Results and discussion

The above analysis made it possible to distinguish three groups of dilatometric effects associated with thermal expansion as applied to heating a sample (in the annealed state) of the 15Kh13N2MFB alloy

[7]. The dilatometric effect of ferromagnetism in the form of an increase in the size of the sample takes place at temperatures below the Curie point ( $780^{\circ}\text{C}$ ). Deviations from the background curve reflect the magnetostrictive effect. The existence of two mutually exclusive processes of changing the sample size in the temperature range of the  $\alpha \rightarrow \gamma$  transformation takes place at higher temperatures.

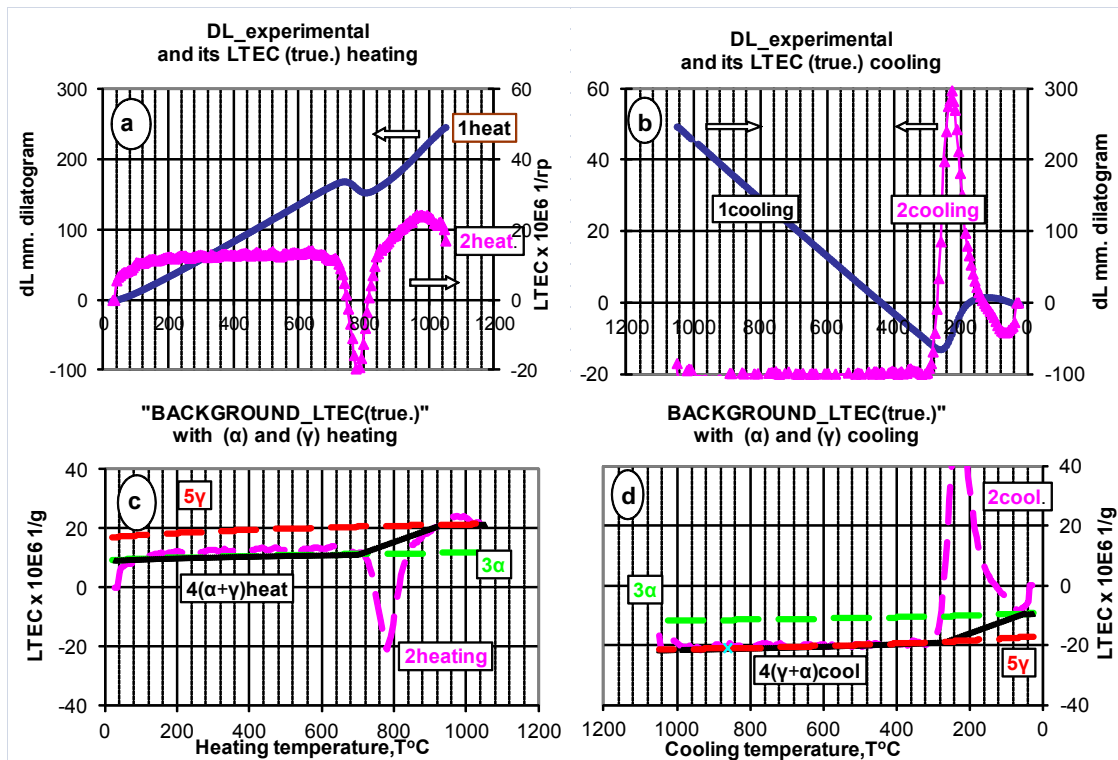
Firstly, this is an intensive reduction in the size of the sample in accordance with generally accepted ideas. Secondly, a small effect of an increase in size was revealed, which experimentally does not contradict the process of transition from the “ferritic” level of LTEC to the “austenitic” one. Such a bifurcation is significant and obviously requires close attention.

The initial data were dilatograms of cooling from austenitization temperature  $1050 \pm 10^{\circ}\text{C}$ , (15 min) at a speed of 50 degrees per minute. The same heating and subsequent cooling curves were averaged before further processing. Two initial conditions were used before heating: after preliminary quenching at  $1050^{\circ}\text{C}$ , 0.5 hours, cooling in air and subsequent tempering at  $650^{\circ}\text{C}$ , 2.0 hours/air.

The initial averaged dilatograms of direct measurements are presented in Figure 1a, 1b during heating (1 heating) and subsequent cooling (1 cooling). A “countdown” on the temperature scale was used for cooling.

Differentiation by two adjacent experimental points was carried out in order to obtain the true values of the TEC [4]. The result is presented in the form of temperature curves of changes in the true values of the thermal expansion coefficient of heating (2heating) and cooling (2cooling), respectively (Figure 1a, 1b). The values of the thermal expansion coefficient of cooling were calculated so as to obtain the opposite sign of the change in the thermal expansion coefficient of heating.

The logarithmic lines  $3\alpha$  (Figure 1c) and  $3\alpha$  (Figure 1d) are drawn with reference to the ferritic state of the sample material ‘ferrite level of LTEC’. Similarly, the logarithmic lines  $5\gamma$  (Figure 1c) and  $5\gamma$  (Figure 1d) were drawn, but with respect to the austenitic state of the structure ‘austenitic level of LTEC’.

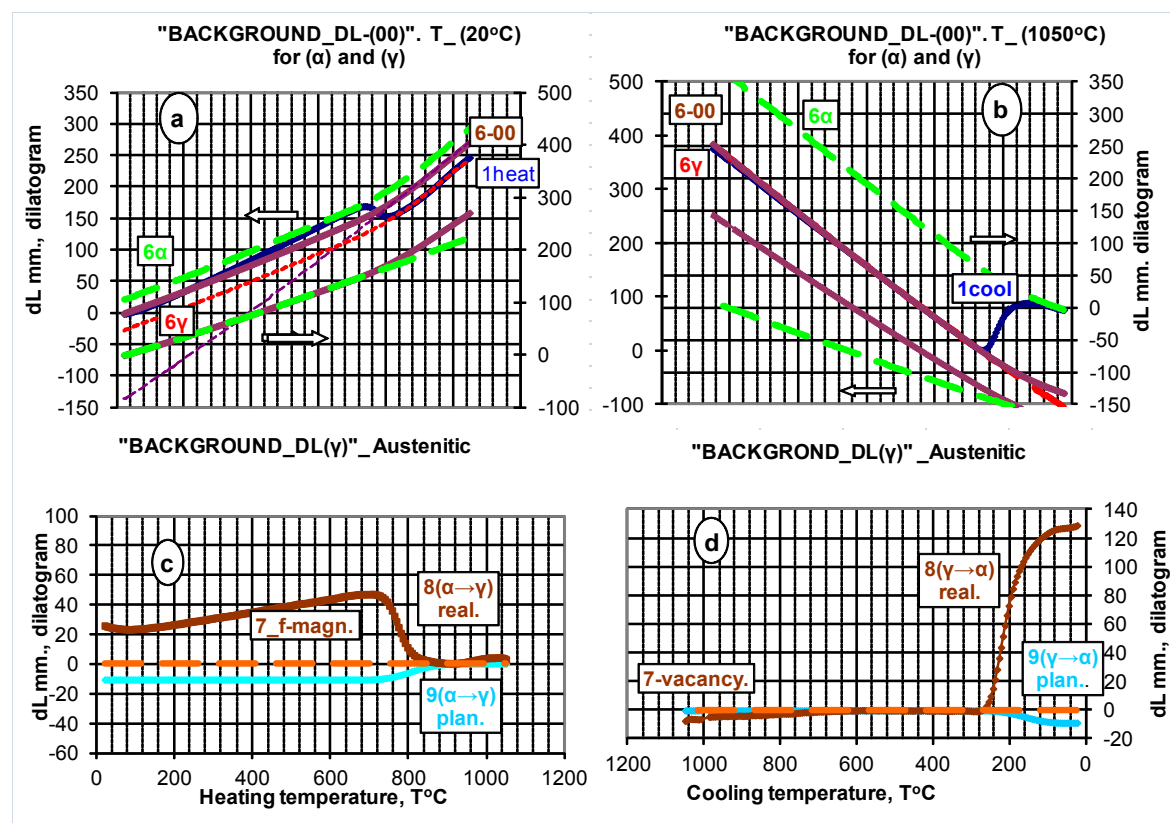


**Figure 1.** An example of processing averaged dilatograms of heating (a, c) and cooling

(b, d, with a countdown of temperature) of 15Kh13N2MFB alloy samples after quenching and subsequent tempering/.

For the ferritic state, this is the temperature range limited by the Curie point (750...780 °C). During cooling, this is the portion of the dilatogram from the minimum temperature of the manifestation of the vacancy correction zone (about 500 °C) up to the temperature of the onset of martensitic transformation (about 300 °C).

In this case, the values of the prelogarithmic factor ( $\Delta \ln$ ) were for the ferritic state ( $\alpha\text{-}\Delta \ln$ ) = 1.65 (heating) and – 1.65 (cooling). For the austenitic state ( $\gamma\text{-}\Delta \ln$ ) = 3.0 (heating) and – 3.0 (cooling). For almost pure iron, the absolute values of such a factor were ( $\alpha\text{-}\Delta \ln$ ) = 1.97 and ( $\gamma\text{-}\Delta \ln$ ) = 3.27 [4, (Figure 2b)]. The introduction of 13 % chromium into the chemical composition of the alloy affected the decrease in the multiplier ( $\Delta \ln$ ).



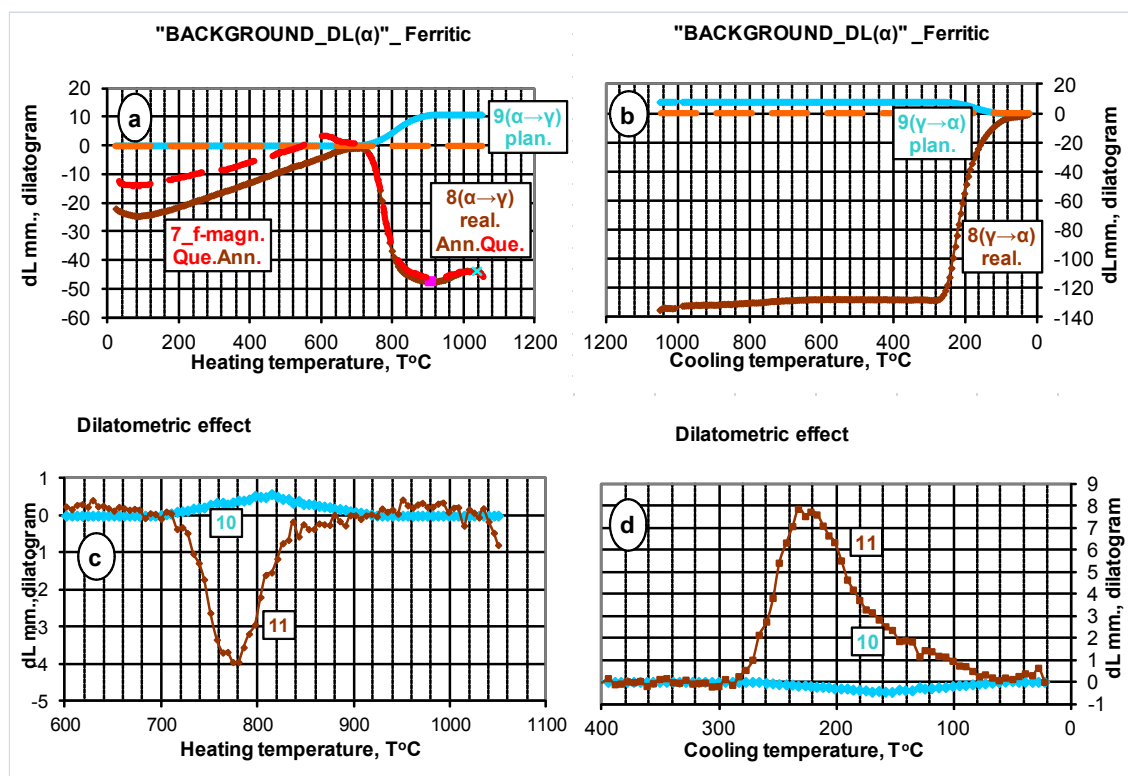
**Figure 2.** Examples of background lines (6-00, a and b) relative to experimental dilatograms (1 heating and 1 cooling). Type of dilatometric effects, reflecting the occurrence of thermal expansion processes (c and d) for the ‘austenitic’ line during heating (c) and cooling (d) of the 15Kh13N2MFB alloy.

It is assumed that broken lines (4 ( $\alpha + \gamma$ ) cooling and 4 ( $\gamma + \alpha$ ) heating) in terms of LTEC should reflect the process of monotonic thermal change in sample size in its pure form. Moreover, the sections of the experimental LTEC curves (curves with index 2) that do not coincide with the course of the constructed ‘total’ background curves (with index 4) are a result of thermal resizing in the material (Figure 1). It is these areas that are ignored in the process of forming the ‘total’ background lines of the LTEC (with an index of 4 in Figure 2).

The next step is the process of numerical integration of the obtained ‘BACKGROUND-LTEC (total) curves’ in order to obtain ‘DL-BACKGROUND’ curves with the dimension of experimental dilatograms. The result of this operation is shown in Figure 2 curves 6-00.

It should be noted that the result of numerical integration of the 4 ( $\alpha + \gamma$ ) cooling and 4 ( $\gamma + \alpha$ ) heating lines is a series of parallel curves of type 6-00, the location of which is regulated by the value of the free term of integration. In curves 6-00 in Figure 2, their location corresponds to the situation when the 'DL-BACKGROUND' line is drawn for the variant of coincidence of the values of the free integration terms when restoring the experimental dilatogram. In this case, the sought-for difference between the experimental dilatogram and the 'DL-BACKGROUND' line has a complex form, making it difficult to clearly identify the distinguished dilatometric effects of structural and phase transformations.

To accurately identify these effects, it is proposed, by selecting the value of the free term of numerical integration, to combine the 'DL-BACKGROUND' line with the characteristic sections of the experimental dilatogram. For Figure 2 a is the beginning of heating (line 6- $\alpha$  'ferritic variant'), and for Figure 2 b – the beginning of cooling (line 6- $\gamma$  'austenitic variant'). These situations correspond to the single-phase state of the sample structure outside the temperature ranges of structural and phase transformations.



**Figure 3.** Dilatometric effects of thermal expansion (a and b) for the 'ferrite version' of the DL-FON line arrangement during heating (a) and cooling (b) of the 15Kh13N2MFB alloy. The result of preliminary differentiation of the pair effect sections during heating (c) and cooling (d) curves.

So, for Figure 2 a, 2 b, the 6- $\alpha$  zero line is drawn according to the 'ferritic version', through the Curie point upon heating and at the time of the 100 % ferrite phase upon cooling at the end point of the martensitic transformation. Accordingly, the 6- $\gamma$  zero line is drawn according to the 'austenitic version'. We combine the line with the regions of the purely austenitic state of the sample structure, above the temperature range of the conversion of ferrite to austenite during heating and in the absence of vacancy formation from about 500 °C to the onset of martensitic transformation upon cooling. In these cases, the difference between the DL-FON zero line and the experimental dilatogram reflects the structural and phase transformations represented by curves 7, 8, in Figure 2 and 3.

In addition to curves 7 and 8, Figure 2 and 3 show a curve with index 9 ( $\alpha$ - $\gamma$ ) expected. The formation of these curves is illustrated by the lower groups of lines in Figure 2 a and 2 b with dashed sections of curves. It is this difference that should reflect the 'expected' effect from the thermal change in the sample size at the recrystallization. At the same time, a large dilatometric effect 8 ( $\alpha$ - $\gamma$ ) appears additionally to this effect as an additional result of the phase transformation in its temperature range.

#### 4. Conclusion

The zero line as the base line separates the 'expected' effect of purely thermal expansion of the sample material from an additional set of dilatometric effects. Effects are represented by mirrored paired sections of 8-expect curves and 9th observation (Figure 2 and 3). This feature allows us to consider that this effect is not random.

A free integration term can establish various representations of these effects. In particular, for the 'ferritic' and 'austenitic' options. Moreover, when heating, it is preferable to use the 'ferritic' version (Figure 3 a), and when cooling, it is 'austenitic' (Figure 2 d). For both options, in the case of analysis by differentiation of the segments reflecting this pairedness, identical curves are obtained in the form of maxima presented in Figure 3 c and 3 d by curves with indices 10 and 11, respectively.

The dilatometer, in this case, plays the role of a DTA device, which has its own sample as a temperature sensor, detecting local changes in the temperature of the sample under the influence of absorbed or released heat of the structural or phase transformation itself against the background of general thermal effects by changing its size. In this case, the recorded maximum of the true LTEC curve should mark the temperature corresponding to the maximum rate of heat generation/absorption in a situation of an equal number of components involved in the transformation. The position and dimensions of the base of the maximum LTEC should mark the beginning and end of the temperature range of the investigated transformation.

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